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Non-monotonic material contrast in scanning ion and scanning electron images

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ABSTRACT

30 keV Ga^+ focused ion beam induced secondary electron (iSE) imaging was used to determine the relative contrast between several materials. The iSE signal compared from C, Si, Al, Ti, Cr, Ni, Cu, Mo, Ag, and W metal layers does not decrease with an increase in target atomic number Z_2, and shows a non-monotonic relationship between contrast and Z_2. The non-monotonic relationship is attributed to periodic fluctuations of the stopping power and sputter yield inherent to the ion–solid interactions. In addition, material contrast from electron-induced secondary electron (eSE) and backscattered electron (BSE) images using scanning electron microscopy (SEM) also shows non-monotonic contrast as a function of Z_2, following the periodic behavior of the stopping power for electron–solid interactions. A comparison of the iSE and eSE results shows similar relative contrast between the metal layers, and not complementary contrast as conventionally understood. These similarities in the contrast behavior can be attributed to similarities in the periodic and non-monotonic function defined by incident particle–solid interaction theory.

1. Introduction

Scanning focused ion beam (FIB) and electron beam instruments (FIB and SEM, respectively) instruments can be used for specimen preparation, direct 2D and 3D materials characterization, and nanoprototyping [1]. Commercial FIB columns typically use Ga^+ ions originating from a liquid metal ion source. One of the advantages of FIB-based procedures is that ion-induced secondary electrons (iSE) or secondary ions (SI) can be collected to form an image such that the ion beam can be precisely placed e.g., for cross-section SEM or transmission electron microscope (TEM) specimen preparation or other processes. Resolution, contrast, and signal-to-noise determine the quality of an ion image, but sputtering typically limits resolution for small features [2]. An image resolution of ~5 nm is common for a Ga^+ FIB operating at 30 kV and ~1 pA beam current [2]. It is well known that the SE yield for ion–solid interactions is larger compared to electron–solid interactions, and scanning ion beam SE (iSE) images also provide unique imaging contrast [3].

1.1. Similarities between ion-induced electron emission and sputtering

Hofer provided a review of ion-induced electron emission from solids concentrating on emission from heavy ($Z > 2$) ions with energy < 100 keV [4]. Hofer discussed and emphasized the similarities between ion-induced electron emission yields and

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physical sputtering yields. The similarities include (i) the energy and angular distributions, (ii) the crystallographic channeling influence, and (iii) the dependency of yields on the projectile energy.

Ion channeling behavior is a well known phenomenon where the ion–solid interactions change drastically with crystal orientation affecting the sputter yield and imaging contrast [5]. In general, darker grains sputter more slowly than brighter grains. These dark grains are consistent with ion trajectories parallel or nearly parallel to low index crystallography planes where ions will travel long distances prior to losing energy and interacting with the target. Since most of the ion–solid interactions occur deeper in the sample, the sputter yield is lower and the grayscale signal is darker. Grains, which are oriented greater than the critical angle to the ion trajectory will cause the ions to interact closer to the surface, losing energy more quickly, yielding a brighter signal. The direct iSE observation of either grain boundaries or interphase interfaces from either polycrystalline materials or multi-phase materials is quite useful and directly interpretable. As is evident, crystallographic effects have similar influences on both the sputter yield as well as the electron emission.

A contrast effect in materials has been observed whereby severe iSE imaging or sputtering of a surface can result in the reorientation of grains and/or the precipitation of a Ga-metal intermetallic due to Ga implantation [6,7]. These new grains tends to grow with a preferred orientation, become resistant to additional sputtering, and result in a dark iSE image. The growth of these new dark grains is easily observed in real time during excessive ion imaging with large doses. This phenomenon shall be referred to as ion beam induced microstructural alteration (IBIMA). Both ion channeling and IBIMA discussed above draw obvious parallels between contrast behavior (i.e., secondary electron emission) and sputtering behavior (i.e., particle emission) as discussed by Hofer [4].

1.2. Recent observations of FIB vs. SEM material contrast

A recent review of iSE material contrast is summarized below [8]. The first observation is that iSE images yield complementary (i.e., reverse) contrast to electron induced SE (eSE) images; i.e., materials that appear bright in eSE images appear dark in iSE images [9]. Si, Al, Cu, Ag, and Au targets were analyzed and it was reported that the atomic number dependence of the SE yield results in opposing image contrast between iSE and eSE images [9]. The second and related observation is that iSE contrast decreases as the atomic number of the target, Z_2, increases [10,11]. Monte Carlo simulations of ion-induced kinetic electron emission were used to predict the iSE yield for Al, Cu, and Au, which also showed a decrease in iSE yield with an increase in Z_2 [11,12].

The observations in the paragraph above are in conflict with numerous earlier reports on electron emission from surfaces showing non-monotonic behavior directly attributed to the periodic influences of ion–solid interactions such as the stopping power [13–16]. Recent preliminary observations also show non-monotonic contrast behavior [17–19]. Additional conclusive evidence is presented and explained fully herein.

1.3. Ion–solid interactions

In this paper, we revisit the SE contrast obtained from FIB images and make use of Monte Carlo TRIM simulations [20] of ion–solid interactions to explain the observed contrast behavior. The basics of ion–solid interactions are reviewed below [21–24]. The energy loss per unit distance (dE/dx) of any charged particle within a target is referred to as the stopping power,

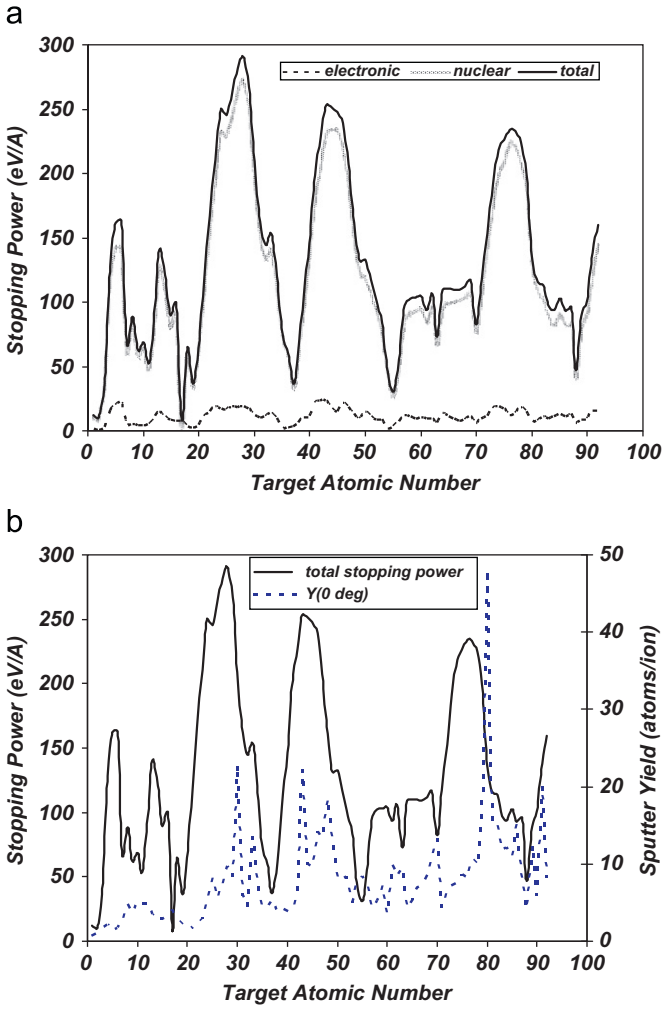

Fig. 1. (a) TRIM determined nuclear, electronic, and total stopping power of 30 keV Ga^+ (Z_1) as a function of target atomic number Z_2. (b) Total stopping power and the sputter yield, Y, for 30 keV Ga^+ ions at 0° incidence as a function of atomic number superimposed on the same graph.

$(dE/dx) = (dE/dx)_n + (dE/dx)_e$, where $(dE/dx)_n$ is due to nuclear collisions and $(dE/dx)_e$ is due to electronic collisions. Nuclear elastic collision terms are calculated assuming conservation of momentum as well as interatomic potentials between ion/target collisions. Electronic collisions involve inelastic collisions between the incident ion and the bound electrons in the target. The electron cloud of the ion may also be involved in collisions. The TRIM determined nuclear, electronic, and total stopping power of 30 keV Ga^+ (Z_1) as a function of target atomic number Z_2, are plotted in Fig. 1(a). Note that the electronic contributions are negligible across the periodic table for the heavy ion example of $Z_1 = 30$ keV Ga^+ ions and therefore the predominant stopping mechanism is controlled by nuclear collisions for this conventional FIB imaging case. Note that the stopping power for each element follows the bonding trends associated with its particular group when moving from left to right across the periodic table yielding a non-monotonic behavior across the entire periodic table. There is an apparent anomaly observed in the peak heights among the elements $Z_2 = 58$–71, which corresponds to the lanthanide series of elements. The smaller stopping power values observed for the physical properties among the lanthanide group are caused by population of the 4f shell in the absence of the covalent bonding contribution of the 5d shell. It is the covalent character of the d shells that imparts the exceptionally high